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IS 1448-131 (1988): Methods of test for petroleum and its products, Part 131: Determination of silicone in petroleum coke [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 131]

DETERMINATION OF SILICON IN PETROLEUM COKE

UDC 665·777·4 : 543 [546·28]

1. SCOPE

1.1 This standard describes an analytical procedure for the determination of silicon in petroleum coke.

2. APPARATUS

2.1 **Platinum Crucibles with Cover** — 30 ml and 150 ml capacities.

2.2 **Volumetric Flasks** — 1 000 ml, 500 ml and 100 ml capacities.

2.3 **Pipettes** — 2 ml, 5 ml, 10 ml, 20 ml and 25 ml capacities.

2.4 **Muffle Furnace** — Capable of controlling between 600 and 1 200°C with regulator.

2.5 **Spectrophotometer or Absorptiometer with 1 cm Cells and Suitable Filters** — Any instrument may be used.

2.6 **Polyethylene Bottles** — 500 ml capacity.

3. REAGENTS

3.1 **Fusion Mixture** — Mix thoroughly 250 g of anhydrous sodium carbonate with 100 g of boric acid powder. Transfer the mixture into a glass stoppered bottle and store in a desiccator.

3.2 **Acid Ammonium Molybdate Solution (10 Percent)** — Dissolve 25 g of ammonium molybdate [$(NH_4)_6 MO_7 O_{24}, 4H_2O$] in 200 ml of distilled water in a borosilicate glass beaker. Add 25 ml of 1 : 1 sulphuric acid. Transfer the contents of the beaker into a 250 ml volumetric flask and make up to the mark with silica-free distilled water. Mix well and store in a polyethylene bottle.

3.3 **Reducing Solution** — Dissolve 3·5 g of sodium sulphite ($Na_2SO_3, 7H_2O$) in 50 ml of distilled water. Add 0·75 g of 1 amino-2-naphthol-4 sulphonic acid and stir until dissolved. Dissolve 45 g of sodium metabisulphite ($Na_2S_2O_5$) in 400 ml of distilled water, add this to the above solution and mix thoroughly. Make up to 500 ml mark, mix again and transfer the solution into a 500 ml polyethylene bottle preferably covered by a sheet of black paper. Keep in a dark place. If properly kept this solution will last for 3 to 4 weeks.

3.4 **Tartaric Acid Solution** — Dissolve 50 g of tartaric acid in about 250 ml of distilled water in a 500-ml volumetric flask. Shake to dissolve and make up to the mark. Mix well and transfer the contents to a polyethylene bottle to be kept in a dark place.

3.5 **Dilute Sulphuric Acid** — Mix one volume of concentrated sulphuric acid with nine volume of distilled water to make 1 : 9 solution.

3.6 **Stock Silica Solution** — Dissolve 5·07 g of sodium metasilicate nona-hydrate ($Na_2SiO_3, 9H_2O$) in recently boiled and cooled distilled water and dilute to 500 ml. Transfer the contents to a polyethylene bottle tightly covered. This solution should contain 1 mg silicon (Si) per ml of solution. Determine the actual silica content of the stock solution as in **3.6.1**.

3.6.1 Take 100 ml of the stock silica solution, add 20 ml of hydrochloric acid and evaporate to dryness in platinum crucible over a water bath. Dry in an oven. To the residue add another 10 ml of hydrochloric acid followed by 50 ml of hot distilled water and filter the suspension so obtained through an ashless medium texture Whatman No. 40 (or equivalent) filter paper. Wash the dish and the

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filter paper with very dilute hydrochloric acid followed by distilled water till the filtrate is free from chloride as indicated by silver nitrate on a drop of filtrate. Set aside the filter paper in a platinum crucible for drying at 110°C. When dry, ignite to constant mass at 1 200°C. Weigh accurately and record the mass. Then add a drop of distilled water to moisten the residue followed by 4 drops of 1 : 1 sulphuric acid and 10 ml of hydrofluoric acid from the reagent container. Slowly evaporate avoiding, any sputtering and when dry, ignite at 1 200°C to contant mass. Weigh accurately and record the mass. Subtract the mass of the crucible and contents after hydrofluoric acid treatment from the mass before treatment and calculate the milligrams per litre of silicon (Si) in the solution. If this is different from the calculated mass of silica in mg/l of the stock solution then adjust volume of the solution or in the standards.

3.7 Standard Silica Solution — Take 10 ml of the stock solution (*see* **3.6**) in a one litre volumetric flask and add 900 ml of silica-free distilled water. Mix and make up to 1 litre. Mix again and keep aside for 10 minutes. Transfer the contents to 500-ml polyethylene bottle having tight fitting stopper and discard the balance. One millilitre of this solution contains 10 μg or 0·01 mg of silicon (Si).

3.7.1 Transfer 5 ml, 20 ml and 25 ml of the solution under **3.7** to different 100 ml volumetric flasks using pipettes. Keep temperature around 20 to 25°C if possible. Dilute each aliquot with distilled water to nearly 50 to 60 ml. Mix and add 1·5 ml of ammonium molybdate solution to the first flask and mix again. Allow to stand for 5 minutes and add 4 ml of tartaric acid followed by 1 ml of reducing solution in quick succession. Mix again and dilute the contents to 100 ml mark. Mix the solution thoroughly and proceed to the next flask. Complete the lot and set aside for one hour. All measurements of the reagents shall be done by pipettes. At the expiry of one hour read percent transmittance at a wave length between 765 to 820 nm on the spectrophotometer previously set at 100 percent transmittance on distilled water. Run a blank using the exact quantities of chemicals except the silica solution and measure the transmittance. Plot milligrams of silica in the final developed solutions against photometer readings on a graph paper.

4. PROCEDURE

4.1 Ash 5 to 10 g of petroleum coke passing through 250 micron sieve in a 30 ml platinum crucible at 750°C over-night. Cool and moisten the ash with a drop of methanol to test for unburnt coke. Ash to constant mass and if there is any difficulty the ashing can be done at 950°C. Weigh the ash.

4.2 Add about 2 g of fusion mixture to the cooled platinum dish containing ash and mix thoroughly using a glass rod with a rubber policeman tip. Sweep back any particles on the glass rod with a fine camel hair brush. Place another 2 g of fusion mixture to cover the sample. Cover the dish with the platinum lid and heat over a gas flame at low heat. Take care to avoid any sputtering and complete fusion at dull red heat for 10 minutes. Cool the crucible with the cover on.

4.3 Add 40 ml of sulphuric acid (*see* **3.5**) to a 250 ml tall form borosilicate beaker. Rinse down into the beaker the lid of the platinum crucible with distilled water and keep aside. Rinse the outside of the crucible with distilled water and gently place the crucible in the beaker. Add enough distilled water to cover the crucible and allow the melt to dissolve completely. The beaker may be covered with a watch-glass. Remove the crucible from the beaker after half an hour, rinse thoroughly with minimum water and boil the solution carefully. Cool and transfer the solution to a 500-ml volumetric flask and wash the beaker at least three times adding the washings to the flask. Make up to the mark with distilled water, mix thoroughly and preserve the solution in a polyethylene bottle for subsequent analysis.

4.4 Pipette 10 ml of the sample solution to a 100-ml volumetric flask, add 1·5 ml of acid ammonium molybdate solution, allow to stand for 10 minutes. Pipette 4 ml of tartaric acid solution followed by 1 ml of reducing solution. Mix well and make up to the mark with distilled water. Again mix and keep aside. The *p*H of this solution should be about 1·6.

4.5 Run a blank simultaneously through the entire procedure using fusion mixture, other chemicals and dilution exactly the same way excluding the sample. Take 10 ml for estimation and keep the remaining solution in a tightly closed polyethylene container for the estimations.

4.6 Measure the transmittance within 765 to 820 nm for the blank and the sample. Deduct the reagent blank and calculate net mg silicon (Si) in the aliquot.

5. CALCULATION

5.1 Silicon (Si), percent by mass

$$= \frac{\text{Net mg silicon in aliquot}}{1\,000} \times \frac{\text{Aliquot factor} \times 100}{\text{Mass of the sample}}$$

6. PRECISION

6.1 Analytical laboratories have reported a precision of 0·01 to 0·02 percent at 0·05 to 0·15 percent level which covers amply the maximum permissible silica in a calcined coke.

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